# PROGRESS TOWARD ROLL PROCESSING OF SOLAR REFLECTIVE MATERIAL

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#### **ABSTRACT**

The goal of this work is to demonstrate that high performance solar reflective material can be produced in a roll format using vacuum deposition techniques. The material consists of a multilayer thin film stack on a substrate. The essential feature of the film stack is an alumina film several microns thick deposited over a silver film. The alumina film is deposited under high vacuum using a physical vapor deposition technique called ion beam assisted deposition (IBAD). The alumina film is optically transparent, scratch resistant, and durable. Its purpose is to protect the silver film and maintain high optical reflectance.

Work on this reflective material began five years ago. Samples were produced on polyester substrates by batch coating. Those samples showed excellent durability in accelerated testing at the National Renewable Energy Laboratory (NREL) in Golden Colorado. The principal limitation to commercialization of the process was a low alumina deposition rate. Over the past three years the alumina deposition rate has been increased from 1 nm/s to 20 nm/s.

The next step is to produce material in a roll format. A roll coater has been built to process 30.48-cm wide rolls of solar reflective material. A steel strip with a highly specular surface finish will be used as the web material. The advantage of this material compared to polyester is that it withstands a higher process temperature and lowers final product installation costs. A major technical challenge will be to reproduce high reflector durability in a continuous, high rate process.

### INTRODUCTION

Concentrating solar power systems use large solar reflectors to concentrate sunlight to generate electricity. The widespread application of concentrating solar power generation depends in part on developing a durable, lowcost reflector. NREL working jointly with Sandia National Laboratories as Sun ♦ Lab, is developing reflectors for the U.S. Department of Energy Concentrating Solar Power Program. The goals are specular reflectance above 90% for at least 10 years under outdoor service conditions and a large-volume manufacturing cost of less than \$10.8/m<sup>2</sup> (Short, 1988). Currently, the best candidate materials for solar reflectors are silver-coated thin-glass, and silveredpolymer films. Polymer reflectors are lighter in weight, offer greater system design flexibility, and have the potential for lower cost than glass reflectors. However, silvered polymer has several limitations, includingrelatively high cost, a lifetime less than 30 years, and poor adhesion between the silver and the polymer on exposure to water (Schissel, 1995). Silvered thin (1.0 mm thick) glass is durable but has relatively high cost, fragility in shipping and handling, and the availability of only a single U.S. manufacturer. These materials may meet the current reflector goal, but it is uncertain whether they can meet a longer lifetime goal of 30 years of outdoor service.

Over the past five years Sun Lab has funded SAIC to develop a promising low-cost "ultra-thin glass" reflector combining the best of both thin-glass and silvered-polymer reflectors. The reflector consists of a polymer substrate coated with a copper layer, followed by a layer of silver, and finally by a protective optically transparent alumina top

coating. Earlier, this basic design was recognized as having high potential, but the difficulty has been to find a durable top coating. The alumina top coating is essential to sustain high reflectance in outdoor service. The coating is deposited by IBAD to produce the desired dense morphology (Vetchen, 1990).

In this paper we describe the fundamentals of producing solar reflective material by IBAD. We present the cost and material design goals that underlie the technical effort. We report our progress on increasing the alumina deposition rate and describe the roll coater constructed at SAIC.

# FUNDAMENTALS OF PRODUCING SOLAR REFLECTIVE MATERIAL BY IBAD

The solar reflective material under development at SAIC is a multilayer coating stack consisting of alumina/silver/ copper/substrate (Fig. 1). The concept is to use a silver film protected by a transparent alumina coating to achieve high reflectivity across the solar spectrum. In most experiments the substrate material has been a polyethylene terephthalate film (PET,  $76.2~\mu m$  thick). In that case a copper film is needed to block UV light transmitted through the silver. If copper were not present, the UV light would break down the PET and cause film delamination. Chrome plated steel is being evaluated as a substitute for PET as a roll coating substrate. In that case a copper back layer may not be necessary.

The three layers of the coating stack are deposited under high vacuum sequentially without breaking vacuum. In this way the silver film is never exposed to air, and high reflectivity is maintained. The metal films are deposited by condensation from vapor that is created by thermal evaporation from crucibles. The source of heating is an electron beam at 10 kV directed into the crucible. A beam current of 100 mA is sufficient to generate a condensation rate of several nm/s onto a substrate located 40-cm away. The chamber pressure is typically low 10<sup>-4</sup> Pa. Deposition rates over 100 nm/s can be easily achieved. Since a high optical density is achieved by a metal film only 100 nm thick, copper and silver can be deposited in seconds.

Alumina is also evaporated by electron beam heating, but a higher temperature is needed to reach the same evaporation rate because it is a refractory material. When the deposition rate is only 1 nm/s at a substrate 40 cm away, the evaporation source is white hot. In our chamber, 8 to 10 kW of power is needed to reach 20 nm/s. At this rate the black body radiation from the evaporator is a significant heat load on the substrate. The condensation energy of alumna also contributes to the heat load. Under these conditions, an uncooled PET substrate is quickly damaged. Higher deposition rates would correspond to even higher heat loads.

Another challenge is that alumina partially decomposes upon vaporization. The vapor cloud consists of molecular alumina fragments that are depleted of oxygen, which appears in the vacuum chamber as a background gas. A coating formed from alumina suboxide is brown. To be optically transparent the coating must be fully oxidized. We accomplish this by two methods. First, oxygen gas is injected into the chamber directly over the molten alumina. A higher partial pressure of oxygen reduces the fraction of molecular alumina that is in a suboxide form. Second, an ion beam is directed onto the substrate during deposition. When the beam contains oxygen, it reacts with suboxide to form fully oxidized alumina. As a secondary benefit, the energetic ion beam increases the density and hardness of the coating.

The alumina coating is not only more difficult to deposit than silver but also more time consuming. The reason is not only that the deposition rate is slower, but also that a thicker layer is needed. Testing at NREL showed that the coating should be 2-4 microns thick to protect silver (Kennedy, 1997). This means to deposit a 4-micron thick coating at 20 nm/s, over three minutes is needed.

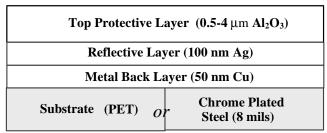


Fig. 1. Structure of solar reflective material.

# **PROJECT TECHNICAL GOALS**

Five years ago the Material Systems Laboratory (MSL) at MIT developed a cost model to describe coating substrates by IBAD (Smilgys, 1996). The model broke down the material production into unit processes, and estimated the cost for each process. The model included variable costs such as materials, direct labor, and energy, as well as fixed costs such as equipment, installation, maintenance, amortization, and real estate. The model allowed prediction of how changes in input parameters affect unit production costs.

The largest contribution to cost is the primary roll coating equipment, adapted from traditional metallization lines in the roll coating industry, along with auxiliary equipment, installation and maintenance. MSL assumed IBAD deposition equipment consisting of one bank of electron beam evaporators and two banks of cold cathode ion sources in a cooled drum roll coater capable of handling a web 107-cm wide, with a web speed below 30.5 m/h. Capital equipment and labor costs accounted for more

than 60% of the total reflector production cost.

The model showed that the deposition rate and thickness of the alumina coating strongly influence the unit cost of solar reflector material. To calculate the minimum deposition rate needed to be cost effective, a "best case scenario" was used. In this case a single roll coater was assumed to operate at maximum capacity with no downtime and 100% yield. Figures 2 and 3 summarize the effect of deposition rate and production volume on unit cost for material with a 2 or 4 micron thick alumina coating. When the alumina thickness is 2 µm, the deposition rate must be at least 30 nm/s before the cost is \$10.8 /m<sup>2</sup>. When the alumina thickness is 4 µm, the deposition rate must be at least 60 nm/s before the cost is \$10.8/m<sup>2</sup>. calculations assumed a total annual production of 0.5 million square meters. Larger annual production volumes did not significantly affect the cost.

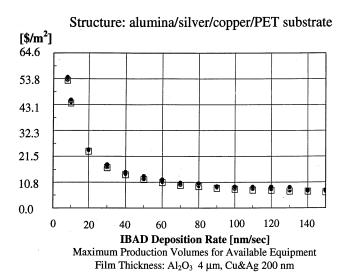


Fig. 2. Analysis of unit cost to produce solar reflective material using roll-coating equipment. Four annual production volumes are analyzed: 0.5, 1.0, 1.5, and 2.0 million sq. meters. The alumina coating thickness is  $4 \mu m$ .

Five years ago the alumina deposition rate was 1 nm/s, and four 15.2-cm square samples could be produced in a single batch. The MSL model established two technical goals that have guided our technical work since that time: (1) increase the alumina deposition rate to 30 nm/s, and (2) transition the IBAD process to a roll coating format. Our progress on the first goal is chronicled in the next section.

## **REVIEW OF TECHNICAL PROGRESS**

## 1. Small Box Coater

The first samples of solar reflective material were

# Structure: alumina/silver/copper/PET substrate

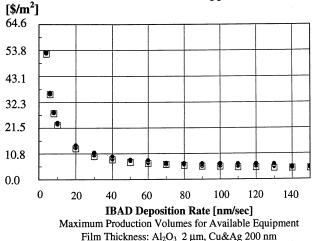


Fig. 3. Analysis of unit cost to produce solar reflective material. The alumina coating thickness is  $2 \mu m$ .

produced in 1995 in a 66-cm wide box coater. The alumina deposition rate was 1 nm/s, and up to four 15.2-cm square samples could be produced in a day. The samples were mounted on a planetary fixture in the upper half of the coater. The planetary moved the samples in a horizontal circle. A cold cathode ion source and a four-pocket e-beam evaporator were located in the bottom half of the chamber. A cooled sheet of copper separated the two halves of the coater. A single opening cut into the sheet passed evaporant flux and the ion beam. The size of the opening allowed only one sample to be exposed at a time. In this way the samples were kept cool by only intermittently exposing them to the beams.

Twenty-five deposition runs were performed using the box coater. The alumina coating thickness ranged from 0.25 µm to 5.0 µm. The samples were subjected to exposure testing in an Atlas Ci65WeatherOmeter (WOM), a 1-kW solar simulator, and outdoor exposure in Golden. CO at NREL. A single day of testing (24 hours) is roughly equivalent to three times the outdoor exposure in terms of light intensity for the WOM and about five times the outdoor exposure in a wavelength band between 300 and 500 nm for the solar simulator. The details of the sample production, testing, and results have previously been reported (Kennedy, 1997). During weathering, loss in specular reflectance has generally been found to be proportional to loss in hemispherical reflectance. Weathering-induced corrosion of the reflective layer causes loss of hemispherical reflectance much sooner than specularity is lost due to surface effects (soiling, crazing, etc.) of the superstrate or a variety of other mechanisms. Spectral hemispherical reflectance is the performance

parameter that is routinely used because it is relatively easier to measure than specular reflectance and because it is the predominant contributor to loss in specular reflectance during weathering.

Optical durability is characterized by plots of optical performance (% hemispherical reflectance) versus time of exposure under specified environmental conditions (for accelerated test chambers) or location (for outdoor sites). Many such plots exhibit sharp changes or large error bars in hemispherical reflectance. These changes are typically caused by measurements taken in different areas of the sample or by discontinuing a degraded sample. standard practice is to measure roughly the center portion of the sample but sometimes breaks, cracks, and large areas of corrosion necessitate measuring in a different region of the sample. Individual curves are typically an average of multiple samples; when a failed sample is discontinued the average can increase, as the poor performer is no longer included in the average value associated with a particular type of material.

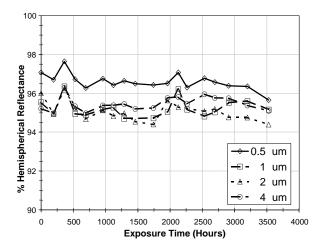


Fig. 4. Solar-weighted hemispherical reflectance of alumina front surface reflectors with various alumina thicknesses (0.5, 1, 2, and 4  $\mu$ m) as a function of solar simulator exposure at 80°c and 80% relative humidity.

The samples were found to maintain high hemispherical reflectance even after 3600 hours of accelerated exposure testing in the solar simulator as shown in Fig. 4. This was significantly longer than any polymer reflector tested in the solar simulators at NREL. The samples also maintained high hemispherical reflectance after four years of outdoor exposure as shown in Fig. 5 and in accelerated WOM exposure testing. The samples were exposed without edge protection and the thinner coated samples began to fail due to flaking around the edges, particularly after snowstorms for the outdoor samples. In spite of this failure, it appeared that the alumina coating

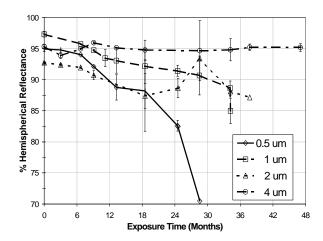


Fig. 5. Solar-weighted hemispherical reflectance of alumina front surface reflectors with various alumina thicknesses (0.5, 1, 2, and 4  $\mu$ m) as a function of outdoor exposure in Golden, Colorado.

continued to protect the silver film from tarnishing. Summarizing, alumina reflectors have excellent durability during accelerated and outdoor weathering. The reflectors with the thickest alumina coating (4  $\mu m$ ) have the greatest durability. The material will require some form of edge protection for long term durability.

## 2. Large Process Chamber

# 2.1 Increase alumina deposition rate to 10 nm/s

By the fall of 1996, the goal was to increase the alumina deposition rate to 10 nm/s and increase the sample size. Since the box coater was inadequate, we began in 1997 to convert a large chamber (cylinder 2.15 m diameter, 3.69 m long) at SAIC to perform IBAD. With a single cryopump it reached a base pressure of low 10<sup>-4</sup> Pa. A 16 cm gridded ion source (1.25 A @ 1 kV), an e-beam evaporator with four 60 cc pockets, and an e-beam power supply (14 kW @ 10 kV) were installed. The evaporator and ion source were located on the bottom half of the chamber. The samples were located in the top half over a set of cooled panels. The evaporant and ion beam passed through an opening in the cooled panels about 20 cm in diameter. This defined the size of the deposition zone.

A major consideration was the greater heat load imposed by a higher alumina deposition rate. Beyond 100°C PET begins to soften, which weakens coating adhesion and lowers material performance. This is a fundamental limitation in the use of PET as a substrate material. We determined that a sample would overheat even if alumina were deposited only one out of every ten seconds with no active cooling.

The intended format for outdoor trials of solar reflective material is a facet on SAIC's Dish/Stirling system. The solar concentrator for SAIC's Dish/Stirling system is the second generation of the Faceted Stretched-Membrane Dish, which was developed under a DOE/Sandia project (SAIC, 1991). The Dish design has 16 facets, each 2.7-meters in diameter. A facet is currently manufactured by welding a stainless steel membrane over a steel hoop like a drum. Thin (<1mm) glass mirrors are manually cut and adhered to cover the membrane. The cost of assembly could be reduced substantially (25-40%) if a steel membrane could be supplied with a reflective finish. The stainless steel foil is not an acceptable substrate for direct metallization because its surface finish is too rough to be a reflector. For this reason we investigated several methods and manufacturers to level the surface of the steel foil. The 0.76-mm thick stainless steel foil (SS) samples were coated with prototype leveling coatings and evaluated. The substrate samples were silvered, stretched to a predetermined tension, and measured at 4, 8, 12, 18, and 25-mrad full-cone angles at 650 nm. The absolute specular reflectance was measured as a function of acceptance angle using a special reflectometer developed at NREL (Susemihl and Schissel, 1987). Most methods were judged as unacceptable due to poor surface specularity, poor adhesion, cost, and feasibility. The two methods that met preliminary requirements were a UV-cured coating and lamination.

Lamination is a process whereby sheets of material are glued together. Two rolls of steel foil 17.8-cm wide were leveled by lamination with PET film by Facile Holdings, Inc., a producer of laminated sheet products. Facile's machinery was designed to laminate PET to aluminum foil, which is much less stiff than stainless steel, but in the event this did not prove to be a limitation. After coating with silver, a PET sheet (0.76 mm thick) laminated to stainless steel foil (0.76 mm thick) (PET/SS) was found to have excellent specularity as shown in Fig. 6.

To reach the 10 nm/s goal, a sample holder was built to actively cool the PET/SS sheet from behind. The holder consisted of a polished aluminum plate 22.9 cm  $\times$  71 cm with a copper sheet cemented to its backside. Copper tubing was soldered to the copper sheet. The PET/SS substrate was adhered to the aluminum plate by a thin layer of thermally conducting paste. During deposition, refrigerated coolant circulated through the tubing and cooled the sample from behind. Since the deposition zone was only about 20 cm wide, to coat a sample 18-cm  $\times$  64-cm, the holder was moved back and forth along its long axis over the deposition zone. Every section of the sample was exposed to deposition about one out of every 10 seconds.

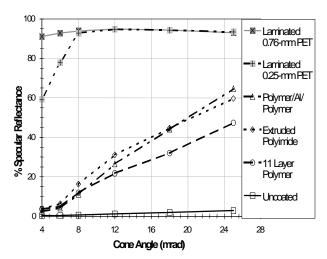


Fig. 6. Specular reflectance at 650 nm of 0.76-mm stainless steel substrates with prototype leveling coatings, silvered, and stretched as a function cone angle in mrad

Over the course of eleven deposition runs, the alumina deposition rate was scaled-up to 10 nm/s. This is the instantaneous deposition rate present when a sample is located over the deposition zone. The coatings were well adhered and transparent. 11.5 nm/s was the maximum rate practical using the four-pocket evaporator because the pocket held too little alumina to sustain a higher rate. If the deposition parameters were properly optimized, the samples maintained high hemispherical reflectance after 14 months of accelerated WOM exposure testing for most increased deposition rates as shown in Fig. 7. In the 7 nm/s deposition rate case, the samples had insufficient ion assist and the durability was unsatisfactory. The 11.5 nm/s samples were not completely oxidized as indicated by the lower hemispherical reflectance. In summary, the alumina reflectors deposited at high deposition rates replicate the excellent durability demonstrated when the alumina was deposited at 1 nm/s.

# 2.2 Increase alumina deposition rate to 20 nm/s

In 1999 the goal was to increase the deposition rate to 20 nm/s. To reach and sustain a higher rate, a rod-fed evaporator (5 cm OD, 25.4 cm maximum length) was installed in the process chamber. The new evaporator held 8.5 times more alumina than a single pocket in the older evaporator, which was retained for evaporation of copper and silver.

Eight runs were performed with a deposition rate greater than 15 nm/s. The sample holder was the cooled aluminum plate described in the previous section (i.e. 22.9 cm $\times$ 71 cm). At first the sample quality was poor due to

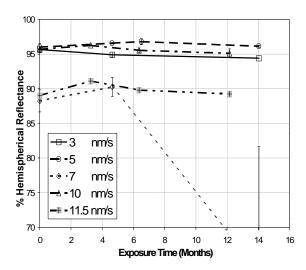


Fig. 7. Solar-weighted hemispherical reflectance of alumina front surface reflectors on prelaminated pet/ss substrates deposited at increasing alumina deposition rates (3, 5, 7,10, and 11.5 nm/s) as a function of WOM exposure at 60°c and 60% relative humidity.

insufficient oxidation of the alumina coating. The samples appeared dark brown with a cracked alumina coating. Up to this time the feed gas to the ion source was a proprietary reactive gas. This gas was found years earlier to produce alumina coatings with fewer cracks than oxygen. Therefore, there was no source of oxygen in the chamber other than oxygen that evolved from alumina evaporation.

To increase coating oxidation two modifications were made to the coating process. First, the ion source feed gas was switched to oxygen. Second, a gas line was plumbed into the chamber to deliver oxygen directly to the alumina evaporator. Shields were also placed around the evaporator to better confine the oxygen over the molten alumina. As discussed earlier, the motivation was to reduce the fraction of alumina suboxide in the vapor..

These two modifications significantly improved the sample quality. The alumina deposition rate for the last sample was 22.5 nm/s on center, and 18.5 nm/ at an edge. The coating was lightly tinted brown at the center, but clear at the edge. The variation in deposition rate from center to edge reflected the variation in evaporation rate from on axis to off axis. In spite of improvement, the process was not optimal for the last sample since there were tensile cracks in the coating. This is an indication of excessive residual tensile stress.

There are practical limits to the alumina deposition rate using IBAD. First, the ion beam assist and the oxygen partial pressure must be scaled with the alumina deposition rate. Both lead to a greater gas load. The highest pressure allowed is low 10<sup>-2</sup> Pa before high voltage arcing occurs. Second, the 16 cm ion source was operating at nearly 80%

of capacity when the rate was 20 nm/s. An increase in pumping and a second ion source will be needed to increase the rate to 30 nm/s.

#### **ROLL COATER CONSTRUCTED AT SAIC**

A roll coater has been constructed at SAIC to produce solar reflective material. The goal is to demonstrate that the coating process is compatible with roll coating, and to produce sufficient material for large-scale outdoor field tests.

The coater consists of a web-handling machine mounted inside the large vacuum chamber. The machine unwinds a web from a payoff reel, wraps it over a cooled drum, and winds it onto a take-up reel. Each reel is driven indirectly through two chains by a DC motor. The reason for indirect drive is to reduce web speed and to develop a higher tension than would be possible with a motor and gear reduction box alone. The signal from an optical encoder is delivered to a PC for speed control of one motor, and measurement of the length unwound. The other motor is operated in regenerative mode to develop hold-back tension. There is not a tensioning spool or dancer to regulate web tension since the web speed is very slow.

The machine is mounted on rails within the chamber with remote axial and lateral motion. The following is a description of the deposition process for a coating stack consisting of two metal films beneath an alumina coating. First, the drum is positioned over the four-pocket evaporator. A fixed length of web is unwound in the first pass to deposit the first film (e.g. Cu). Then the motor direction is reversed, and a second film is deposited in a second pass (Ag). Next the drum is moved sideways to position it over the rod-fed evaporator. The web direction is reversed a third time and the alumina coating is deposited in a single pass.

The reels and cooled drum were made to advance a web 30.48-cm wide. The width was made as wide as possible consistent with achieving uniform coating thickness across the width using a single evaporator. A higher degree of uniformity is possible if necessary by sweeping the web-handling machine side to side during deposition, but this would lower the duty factor.

The web speed is calculated from the desired coating thickness and deposition rate. For instance, when the rate is 20 nm/s, the web speed is 6 cm/min to deposit a 4  $\mu$ m coating in one pass. At this speed a spot on the web spends over three minutes in the deposition zone. These calculations assume a 20 cm length of web is exposed at a time, which is the widest possible using a single ion source. If a second ion source were present on the opposite side of the evaporator, the length of web exposed could be increased. Then the web speed could be increased proportionately.

<u>TABLE 1. SPECIFICATIONS OF WEB-HANDLING MACHINE</u>

web width	30.48 cm	web tension	1-189 N
web speed	1-89 cm/min	drum OD	30.48 cm
reel ID	30.48 cm	drum temp	≥-196°C
max reel OD	36.8 cm		

An important component is the cooling drum around which the web is wrapped during deposition. Heat deposited into the front of the web is removed out the back into the drum. The drum is hollow inside and filled with alcohol. Liquid nitrogen is brought inside the drum through a tube. The flow of liquid is regulated to maintain the alcohol temperature above its freezing point. The rotating axis of the drum is sealed against vacuum by two o-rings. The space between the o-rings is differentially pumped. A heating element keeps the o-rings from freezing. The stationary end of the rotating seal passes liquid and gaseous nitrogen, and two thermocouples. In the event the heat load is much higher or heat conduction through the web is poor, the drum could be filled with liquid nitrogen for maximal cooling.

The coating to level the stainless steel substrate was reexamined in the attempt to remove the PET from the construction and because Facile Coatings was unable to laminate wider rolls and was no longer interested in laminating PET on SS. New methods to level the SS were examined including coatings that had not been initially feasible. In the past we used a laboratory instrument to measure specular reflectance (Susemihl and Schissel, 1987), which is now inoperable because of equipment failure and unavailability of replacement parts. A less accurate unit (Field Portable Specular Reflectometer from D&S Instruments) was used to measure the specular reflectance of the new substrates at 7, 15, and 25-mrad fullcone angles at 660 nm. For these measurements the samples were not silvered or stretched.

A promising alternative to PET is a chrome plated carbon steel strip manufactured by the American Nickeloid Company. The strip is available in 0.92-m wide rolls as thin as 203  $\mu$ m (8 mils) with good surface specularity as shown in Fig. 8. The specularity of the Nickeloid and the laminated PET/SS substrates are comparable when both are coated with alumina and silver layers, even though when neither is coated the Nickeloid substrate is more reflective. The material costs approximately \$7.6/m². In application single strips could be used in a trough design, or multiple strips could be welded together to make a wider sheet (e.g. 3-m wide facet).

We hope for significant improvements in reflector performance when the material is produced in a roll format. First, we speculate that early samples created by batch processing failed due to differential thermal expansion between the various layers of the construction (particularly between the alumina coating and the PET substrate) and/or break down of the PET substrate. A chrome plated steel web should be far more stable in outdoor exposure than PET. Second, unlike PET, thermal damage to a chrome plated steel web is not a factor. Third, when a sample is produced by batch processing, its surface is contaminated by particles due to handling and dust raised during pump down. The alumina coating must be thick enough to bury the largest particles. On the other hand, chrome plated steel web is supplied with a protective cover sheet that will be removed under vacuum prior to deposition. In this way the size and density of surface particles should be much less compared to batch processing. A thinner alumina coating may be adequate to achieve the same level of protection. If true this would reduce cost since the alumina coating is the slowest, most costly step. The durability of the reflector on this substrate needs to be tested and a second cost analysis using a steel web needs to be performed.

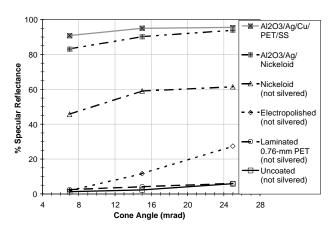


Fig. 8. Specular reflectance at 660 nm of 0.76-mm stainless steel substrates or 203-µm Nickeloid chrome plated carbon steel with prototype leveling coatings, as a function cone angle in mrad.

## CONCLUSIONS

Over the past five years we have made significant progress in developing a new type of solar reflective material. This material has the potential to deliver high performance at a manufacturing cost lower than thin glass. A significant advantage should lie in lowering system weight and installation costs.

Before the material can be commercially viable, we must demonstrate high performance at high rate in a roll format. Only samples on a PET substrate produced by batch coating at low rate have shown high durability to date. We believe a fundamental limitation has been the durability of the PET substrate. The switch to chrome

plated steel has the potential to solve this problem, but the durability of reflector performance on this substrate is still unknown and additional testing is planned.

#### **ACKNOWLEDGMENTS**

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